NON-THERMIONIC SPUTTER MATERIAL TRANSPORT DEVICE, METHODS OF USE, AND MATERIALS PRODUCED THEREBY

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Description

NON-THERMIONIC SPUTTER MATERIAL TRANSPORT DEVICE, METHODS OF USE, AND MATERIALS PRODUCED THEREBY

Technical Field

The present invention is generally directed to deposition of thin films and growth of bulk materials. In particular, the present invention is directed to non-thermionic, plasma-enhanced sputtering techniques.

Background Art

A wide variety of techniques exist for depositing thin films onto substrates in order to achieve desirable properties which are either different from, similar to, or superior to the properties of the substrates themselves. Thin films are employed in many kinds of optical, electrical, magnetic, chemical, mechanical and thermal applications. Optical applications include reflective/anti-reflective coatings, interference filters, memory storage in compact disc form, and waveguides. Electrical applications include insulating, conducting and semiconductor devices, as well as piezoelectric drivers. Magnetic applications include memory discs. Chemical applications include barriers to diffusion or alloying (e.g., galling), protection against oxidation or corrosion, and gas or liquid sensors. Mechanical applications include tribological (wear-resistant) coatings, materials having desirable hardness or adhesion properties, and micromechanics. Thermal applications include barrier layers and heat sinks.

Bulk materials can be used as substrates upon which thin films can be deposited and microelectronic and optical devices can be fabricated.

Thin-film techniques typically entail several sequential process steps. Generally, a source of film-forming material is supplied, the material is transported to the substrate, and deposition occurs on the substrate surface. The material transport step occurs in a contained environment such as a chamber containing a vacuum, one or more gaseous fluids, and/or a plasma medium. Deposition behavior is determined not only by the source and transport factors but also by deposition surface factors. Such surface factors include the substrate surface condition (e.g., surface roughness, contamination, degree of chemical bonding between the surface and the arriving material, and crystallographic or epitaxial parameters); the reactivity of the arriving material (e.g., the sticking coefficient, which provides an indication of the probability of arriving molecules reacting with the surface and becoming incorporated into the film); and the energy input (e.g., substrate temperature, positive-ion bombardment, and chemical reactions). The results of the deposition can be analyzed, and one or more process conditions can be modified as appropriate in order to obtain the specific film properties desired. Process control and monitoring steps are usually carried out at all key points along the process. Post-deposition annealing procedures can also be employed to activate grain growth, alter stoichiometry, introduce dopants, or deliberately cause oxidation.

Deposition processes are broadly delineated into "physical" vapor deposition (PVD) processes and "chemical" vapor deposition (CVD) processes, although some processes might better be characterized as being hybrids of PVD and CVD processes. The source of material supplied to the deposition system

can be a solid, liquid, vapor, or gas. Solid materials must be vaporized in a PVD process in order to transport them to the substrate. Vaporization is accomplished either by employing a thermal technique (e.g., evaporation) or by providing an energetic beam of electrons, photons (e.g., laser ablation), or positive ions (e.g., sputtering). On the other hand, CVD techniques utilize gases, evaporated liquids, or chemically gasified solids as source materials. In both PVD and CVD processes, contamination is a critical factor during the source supply step, as well as in the transport and deposition steps. The source supply rate is also a critical factor, as film properties can vary with deposition rate and, in the case of compound films, with the ratio of elements supplied.

One common PVD process entails thermal evaporation, which is often accomplished by using a twisted-wire coil, a dimpled sheet-metal "evaporation boat," or a heat-shielded crucible. In thermal evaporation, thermal energy alone (i.e., joule heating) is utilized to drive the evaporation, reaction and film structure development. On the other hand, several known deposition processes exist in which the primary source of energy can be characterized as being essentially "nonthermal." In these "energy-beam" techniques, energy is delivered by electrons, photons or ions (usually positive ions) to vaporize the source material, activate the source material during transport, or modify film structure during deposition. Common energy-beam techniques used to carry out vaporization can be broadly categorized as electron-beam, cathodic-arc, anodic-arc, pulsed-laser, ion-beam sputtering, and glow-discharge sputtering processes. Clear differences exist between the first four techniques and the two sputtering techniques. In the first four techniques, electrons (via an electron beam), ions (via an arc) or photons (via a pulsed laser) are directed at the source material in

a narrow beam having a diameter of approximately a few millimeters. Conversely, the ion beams and glow discharges employed in the sputtering techniques cover a much broader area. Additionally, the use of narrow beams leads to intense heating of the source material at the point of impact, so that the vaporization mechanism is thermal even though the energy input is nonthermal. By contrast, vaporization by sputtering involves direct momentum transfer from bombarding ions to the surface atoms of a relatively cool source material.

There are several advantages to using energy beams for vaporization as compared to joule-heated sources. First, virtually any material, no matter how refractory, can be vaporized. In the narrow-beam processes, this is a result of the very high energy density and surface temperature that is achieved. In sputtering, the advantage results from the fact that the bombarding ions have energies far exceeding chemical-bond strengths which typically are only a few electron volts (one electron volt, or 1 eV, will be understood as constituting the energy gain of a particle having one electronic charge upon passing through a potential drop of one volt). Second, in the cases of pulsed-laser evaporation and sputtering, the activated depth of source material can be in the range of only tens of nanometers, which results in stoichiometric (congruent) vaporization of multi-element materials, thereby assisting (albeit not necessarily guaranteeing) a stoichiometric deposit. Third, in all of the energy-beam processes, much of the vapor acquires energy well above the thermal energy of the surface of the source material, and this energy can greatly assist the deposition process. Atoms thermally evaporated by narrow energy beams acquire most of their energy by interaction with the beam in the vapor phase, while sputtered atoms have high energies at the time they leave the surface of the source material. In

the case of ionized vapor, this energy can be further increased by accelerating ions toward the surface of the depositing film, which is accomplished by applying a negative bias to the substrate. Energy can also be directed at the deposition surface through the mechanism of either energetic-atom condensation or ion bombardment, which can result in significant improvement in film adherence and structure.

Figure 1 illustrates the widely used parallel-plate plasma configuration, commonly known as a planar diode and generally designated **10**. Two electrodes, a cathode **12** and an anode **14**, are parallel to each other and spaced apart from each other by a distance or electrode gap **L**. Anode **14** can be at ground or alternatively driven with an RF bias source **16** and associated capacitor **16A** (shown in phantom), and cathode **12** is driven negative by a power supply **18**. A glow-discharge plasma **20** is generated between the two electrodes and confined by a grounded metal vacuum containment wall **22**. The bulk of plasma **20** floats above ground by the plasma potential, and has little voltage drop across it because of its high conductivity relative to that of its sheaths. This means that essentially all of the applied voltage appears across the cathode sheath. This voltage drop results in high-energy ion bombardment of cathode **12** by positive ions **24** and sputtering of cathode **12** as represented by sputtered atom **26**. The cathode voltage drop also sustains plasma **18** by accelerating secondary electrons **28** emitted from cathode **12** into plasma **18** where they initiate a cascade of ionizing collisions. As illustrated, diode **10** can be operated under an applied DC voltage or an RF voltage.

The DC parallel-plate glow discharge typically operates at a pressure in the approximately 3 - 300 Pa range and at an applied voltage of approximately

1000 - 2000 V. The exact pressure range will depend on electrode gap **L** and gas composition. At pressures below the limit, not enough collisions occur before the electrons reach anode **14**. At higher pressures, the discharge tends to switch to the concentrated, low-voltage arc mode, especially at high power. The high voltage of the DC glow discharge is required so that each secondary electron **28** emitted from cathode **12** can produce enough ionizing collisions before losing its energy. A small increase in voltage results in a large increase in current because of the cascade effect, so for good power control a current-regulated power supply is used. To "strike" (initiate) the discharge, it is often necessary to supply a spike of higher voltage, or to adjust pressure to a minimum so that the gas will break down at the voltage available.

Secondary electrons **28** emitted from cathode **12** first cross a "dark space" generally designated **30**. This region is "dark" because an insufficient number of inelastic collisions with molecules occur for any glow from the excited states of the molecules to be observed. The width of dark space **30** may be smaller than that of the sheath at high pressure and low plasma density, or it may be greater in the opposite case. Since the electrons follow the sheath field, which is perpendicular to the cathode surface, the electrons travel in a broad parallel beam and accordingly are known as "beam" electrons. After acceleration, the beam electrons pass into the "negative glow" region of plasma **20**, where they ionize gas molecules and lose their directionality due to scattering. If electrode gap **L** is smaller than the width of the negative glow, the beam electrons are likely to reach anode **14** before undergoing an ionizing collision. Such a discharge is said to be "obstructed," and any further decrease in electrode gap **L** causes a sharp rise in voltage and ultimately extinction of

plasma **20**. The width of the negative glow is roughly equal to the mean free path for ionizing collisions. Undesired discharges along the back of cathode **12** and its voltage lead **32** can be prevented by installing a grounded "dark-space shield" (not shown) along these surfaces.

A mode of plasma-enhanced chemical activation generally known as "reactive sputtering" uses a sputtered source material along with a gaseous one. The gas becomes dissociated in the sputtering plasma and reacts to form a compound film. The parallel-plate plasma configuration of Figure 1 can be used to supply vapor for film deposition by sputter-erosion of cathode **12**, which in this case is termed the "target" material. Often, the plasma is magnetized using a magnetron assembly generally designated **40**, as described hereinbelow. In either case, cathode **12** is bombarded by plasma ions **24** having energies approaching the externally applied voltage, although ion energy is distributed downwardly by scattering in the sheath. Chief effects of the plasma on sputtering process behavior are: (1) reactive sputtering, (2) scattering of the particles by the plasma gas, (3) negative-ion ejection from the target, and (4) resputtering. Resputtering involves the acceleration of plasma ions into the substrate using a negative bias. The resultant resputtering of the depositing film can produce effective planarization of rough topography, and the bombardment can modify film structure in various known ways.

In the technique of reactive sputtering, a reactive gas (e.g., N_2) is added to the sputtering plasma (e.g., argon gas plasma) in order to shift compound-film stoichiometry in sputtering from a compound target, or to deposit a compound film from a metallic target (e.g., Al). Compound deposition by reactive sputtering from a metallic target generally lowers target fabrication costs and increases

target purity as compared to using a compound target, but process control can be more difficult if film composition is critical.

Even at the lowest operable pressure of the DC-diode plasma, there is considerable gas scattering of sputtered particles as they cross the plasma, with consequent loss of their desirable kinetic energy and loss of deposition rate by backscattering. Magnetic confinement is widely used to reduce minimum pressure and thus avoid these problems. Scattering of the sputtered particles also broadens their spread of incident angles at the substrate. Thermalization and spreading together cause a generally undesirable shift in film microstructure from a bombardment-compacted structure (e.g., “Zone T”) to the more porous and weakly bonded structure (e.g., “Zone 1”). Operation at lower plasma pressure using magnetron assembly **40** avoids this problem.

With respect to negative ions ejected from a compound target, when one element has a low ionization potential (e.g., 6 eV) and the other has a high electron affinity (e.g., 2 eV) so that the difference between the two becomes small, it is likely that the latter element will be sputtered as a negative ion rather than as a neutral atom. Negative ions are accelerated into the plasma along with the beam electrons by the cathode sheath field. For pressures above about 1 Pa, the negative ions will be stripped of the extra electron in the plasma. But unless the product of electrode gap **L** and gas pressure is very high, the ion can still cross to the depositing film and bombard it with enough energy to damage or erode the film. When the negative-ion flux is substantial in glow-discharge sputtering of compounds, problems can be encountered at both low and high operating pressures. At low pressure, the desirable kinetic energy of the sputtered particles is retained but negative-ion damage can result. At high

pressure, the undesirable negative-ion energy is dissipated but Zone T film structure can be lost as a result of thermalization and scattering.

When employing a planar-diode plasma configuration to cause sputtering, the beam electrons ejected from cathode **12** must undergo enough ionizing collisions with the gas to sustain plasma **20** before the beam electrons reach anode **14** and are removed there. This requirement places a lower limit on operating pressure, and can be enhanced through the use of magnetron assembly **40**, as illustrated in Figure 1. Magnetron assembly **40** typically includes a central bar magnet **42** and an outer ring magnet or magnets **44** of opposite pole. Magnetron **40** produces a cross-wise magnetic field over cathode **12**. The magnetic field traps the beam electrons in orbits near the cathode surface. As a result, the path lengths of the beam electrons are significantly increased before the electrons finally escape to anode **14** by collisional scattering. Because the paths of the electrons become longer than electrode gap **L**, the minimum pressure needed to sustain plasma **20** is much lower (typically 0.1 Pa rather than 3 Pa) when using magnetron **40** as compared with planar diode **10** without magnetron **40**. At a lower pressure (e.g., 0.1 Pa), the sputtered particles retain most of their kinetic energy upon reaching the substrate, and this energy has advantageous effects on the structure of the depositing film. In addition, deposition rate is increased due to reduced scattering and redeposition of sputtered particles on cathode **12**. Moreover, the beam electrons are utilized more efficiently, with the result that a lower applied voltage (e.g., approximately 500 V) is required to sustain a plasma of a given density, and the voltage increases less steeply with power input as compared to a non-magnetron planar diode configuration. Negative ions can still be a

problem, however. Also, a highly non-uniform erosion pattern appears on the target cathode surface. If negative ions influence the film during deposition, this pattern can become imprinted on the film as it is being deposited on a stationary substrate as a result of the beam nature of the negative ions. However, since the sputtered particles are neutral and are emitted in a generally cosine distribution, the non-uniformity of the deposition rate is less sharply imprinted on the film. It should also be noted that, as in the case of planar diodes, magnetrons can be operated under RF excitation if power is to be coupled through insulating targets.

Referring to Figures 1 and 2, magnetron **40** has a planar, circular configuration. The target material of cathode **12** is a disc, typically 3 - 10 mm thick, and is bonded (such as by soldering, for good thermal contact) to a water-cooled copper backing plate **50**. The water coolant can be deionized to prevent electrolytic corrosion between electrically biased backing plate **50** and a grounded water supply **52**. Cathode **12** is often floated off ground with a ceramic insulating ring (not shown). Containment wall **22** serves as an anode, although grounded shields (not shown) can be added to confine the sputtered material. The cross-wise magnetic field is established by magnets **42** and **44**. Magnets **42** and **44** are connected on the back by an iron "field-return" plate **46** to complete the magnetic circuit and to confine the magnetic field.

Upon igniting plasma **20**, beam electrons emitted from cathode **12** are accelerated into plasma **20** by the electric field of the cathode sheath. The presence of the magnetic field, represented by virtual magnetic field lines **B** in Figure 2, causes the beam electrons to curve into orbits as a result of the Lorentz force, $F = F_E + F_B = q_eE + q_ev \times B$. The radius of the orbit (referred to as

the gyratron, cyclotron or Larmor radius) depends on the strength of the magnetic field and on the electron velocity component perpendicular to the magnetic field. In order for the magnetic field to have an effect on the beam electrons, the pressure must be low enough (typically less than a few Pa) that the electron mean free path is not significantly less than the orbit radius. If this condition is met, the beam electrons are said to be "magnetized" although the ions are not magnetized. Magnetron **40** can operate as a sputtering source at much higher pressures, but in such cases gas scattering dominates the behavior of the beam electrons instead of the magnetic field itself.

Under lower pressure conditions, the beam electrons emitted from the target surface of cathode **12** or created by ionization in the sheath field are accelerated vertically by the electric field and simultaneously forced sideways by the magnetic field. The beam electrons eventually reverse direction and return toward the target. As the beam electrons are thus directed toward the target, they decelerate in the electric field until their direction is again reversed, and the cycle repeats. As specifically shown in Figure 2, the net motion or path of these electrons is a circular drift path, designated **E x B**, around the circle of the target. This drift path is in the direction of the **E x B** vector product. Magnetron **40** is ordinarily designed such that the **E x B** drift path closes on itself so that the beam electrons do not pile up or accumulate at some location.

Additionally, cathodic structures have been developed to enhance processing-scale plasmas such as magnetrons and RF diodes by taking advantage of the "hollow cathode" effect, a phenomenon which generally involves utilizing geometric means to trap secondary electrons emitted from an ion-bombarded target cathode. When a hollow-cathode-type structure is driven

to a very high discharge current, its cathode surfaces heat to a temperature sufficient to cause thermionic emission of electrons, and the local plasma glow discharge will enter the arc mode. A hollow cathode, typically constructed of a refractory material and provided with a local gas supply, can be a useful source of moderately energetic electrons for plasmas.

Referring to Figure 3, a sputter transport device generally designated **60** includes a planar configuration of a magnetron generally designated **62**, a target cathode **64**, a substrate holder **66**, and a substrate **68**, all of which are situated in a containment chamber **70**. A hollow cathode generally designated **72** is provided in the form of a tube **72A** having a tantalum tip **72B**. A gas source (not specifically shown) is connected to one end of hollow cathode **72**, and a small aperture or orifice **72C** is provided at the tip. Aperture **72C** restricts the gas flow and results in a large pressure differential across tip **72B**. The inner pressure of hollow cathode **72** is typically in the range of several hundred mTorr. Electrons are emitted by biasing hollow cathode **72** negatively with respect to the local plasma potential (which is usually the ground potential). A hollow cathode having a diameter of only a few millimeters can be employed to produce an electron current of several to ten amperes. An external heater or a short-term, high-voltage spike is typically used to heat hollow cathode **72** to the temperature required for emission.

In Figure 3, hollow cathode **72** is situated in the fringe region of the magnetic field of magnetron **62** to supply additional electrons to the magnetron discharge. Hollow cathode **72** serves to decouple the current-voltage relation of the diode plasma and allow operation of the plasma at wide ranges of voltage and current, as well as to lower the operating pressure in chamber **70**. Hollow

cathode **72** can operate at 0.1 mTorr, which is below the range of the more conventional magnetron/diode arrangement described hereinabove and illustrated in Figure 1. If conventional magnetron/diode arrangements were to operate at these lower pressures, there would be not be enough gas atoms for efficient ionization by the secondary electrons. The additional supply of electrons from hollow cathode **72**, however, removes this limitation and allows operation at approximately 0.1 mTorr for magnetron arrangements, and approximately 0.5 mTorr for RF-diode arrangements. Such pressures are well into the long mean free path mode, and sputtered atoms or ions move in straight, line-of-sight trajectories without gas scattering.

A magnetron sputter device enhanced with a hollow cathode source capable of emitting a high electron current is disclosed in U.S. Patent No. 4,588,490 to Cuomo et al., the specification of which is incorporated herein by reference. Similar to the apparatus illustrated in Figure 3 of the present disclosure, the invention disclosed in U.S. Patent No. 4,588,490 combines a hollow cathode electron emitting device with a known plasma sputter etching/deposition device, in order to provide additional ionization of the working or background gas during normal magnetron operation and to provide gas ionization at low magnetron energies. The hollow cathode source is provided in the form of a tantalum tube, and is positioned such that it is immersed in the transverse magnetic field near the magnetron cathode target surface, but neither electrically nor physically impedes the magnetron **E x B** drift current. The discharge plasma initiated and maintained within the hollow cathode is thermionic in nature. The hollow cathode is biased negative with respect to plasma potential, which causes thermionic heating of the tantalum tip. The

thermionically emitted electrons become trapped and distributed around the magnetron drift loop by a modified **E x B** effect. These electrons are energetic enough to cause ionization of the background gas and to ionize the argon gas flowing through the tantalum tip. The increased ionization forms a denser plasma, such as dense plasma region **76** in Figure 3, than can be produced by the magnetron alone, which plasma is characterized by a lower impedance that results in increased currents at constant voltage.

While hollow cathode enhanced sputtering devices provide advantages over many of the other deposition techniques described hereinabove, there are still drawbacks with regard to their use, owing to the fact that they are thermionic emitting electron devices. For instance, contamination is still observed to be a problem, particularly since the hollow cathode tip material tends to evaporate and mix with the growing deposition material. Another problem relates to the intense heat produced by thermionic emission, which can damage the growing material.

The present invention is provided to address these and other problems associated with the growth of thin films and bulk materials.

Disclosure of the Invention

The present invention provides a physical vapor deposition (PVD) technique enabled by a novel sputter material transport device to enhance thin-film and bulk material manufacturing processes. The novel transport device is capable of ultra-high deposition and growth rates, making it feasible for growing thick material and increasing throughput in manufacturing processes. The transport device can be used both to grow bulk crystalline materials and to

deposit thin films and epitaxial layers onto bulk substrates. Generally, as compared to other sputter processes, the transport device of the present invention has the advantages of lowered processing pressure, higher deposition rates, higher ionization efficiency, and a controlled processing environment with no contamination. The novel device utilizes an enhanced sputtering process to rapidly deposit both metallic and dielectric materials. This enhancement allows the process to overcome the limitations of conventional PVD techniques.

The device according to the present invention can achieve growth rates in excess of ten times those achieved by any other direct deposition process. As currently tested, the device is capable of depositing single or polycrystalline material at a rate in excess of approximately 60 μm/hr. This high deposition rate allows for high throughput capabilities and the possibility of manufacturing bulk materials in short time periods. The device enables increased growth rates due to the very high ionization efficiencies, which enhance the sputtering process without poisoning the sputtering material. The ability to deposit material at high deposition rates will have many commercial applications, including high-throughput manufacturing processes of thick films of exotic materials. Moreover, high-quality material can be deposited in a cost-effective manner. It is also projected that the device will aid in the commercialization of bulk dielectric and semiconductor materials and will have numerous applications to other materials.

The invention surpasses present technology by offering a non-contaminating method, as implemented by a triode sputtering device, to increase the ionization efficiency and hence the overall deposition rate. The device also has the advantage of a cooler operating temperature than a thermionic hollow cathode configuration, allowing the injector means of the device to be composed

of low-temperature materials, and thus can apply to a broad range of materials as compared to conventional processes. The transport device can increase the deposition rate of the target material and lower the sputtering pressure, thereby enabling a line-of-sight deposition process.

The transport device is capable of growing bulk material such as aluminum nitride, gallium nitride, and other Group III nitrides and related binary, ternary, and quaternary alloys and compounds. The transport device is also capable of depositing metal in deep trenches for the semiconductor industry.

According to the present invention, the transport device includes a magnetron source and a non-thermionic electron (or, in effect, a plasma) injector assembly to enhance magnetron plasma. Preferably, the electron/plasma injector is disposed just below the surface of a cathode target material, and includes a plurality of non-thermionic, hollow cathode-type injector devices for injecting electrons into a magnetic field produced by a magnetron source. The injector can be scaled in a variety of configurations (e.g., circular or linear) to accommodate various magnetron shapes. When provided in the form of a circular ring, the injector includes multiple hollow cathodes located around the inner diameter of the ring.

The novel transport device constitutes an improvement over the previously developed hollow cathode enhanced magnetron sputtering device described hereinabove, in that the device is a non-thermionic electron emitter operating as a "cold" plasma source and can be composed of the same material as its sputtering target. The injector can be manufactured out of high-purity metals (e.g., 99.9999%), thereby eliminating a source of contamination in the growing film. The addition of the injector to the magnetron sputtering process

allows higher deposition rates as compared to rates previously achieved by conventional magnetron sputtering devices. Moreover, the transport device takes advantage of the hollow cathode effect by injecting electrons and plasma into the magnetic field to increase plasma densities without the contamination problem associated with a traditional, thermionic-emitting tantalum tip. As disclosed above, the transport device is further characterized by a decreased operating pressure and an increased ionization rate over conventional magnetron sputtering.

According to one aspect of the present invention, a sputter transport device comprises a sealable, pressure-controlled chamber defining an interior space, a target cathode disposed in the chamber, and a substrate holder disposed in the chamber and spaced at a distance from the target cathode. The target cathode is preferably bonded to a target cathode holder and negatively biased. A magnetron assembly is disposed in the chamber proximate to the target cathode. A negatively-biased, non-thermionic electron/plasma injector assembly is disposed between the target cathode and the substrate holder. In a preferred embodiment of the invention, the injector assembly comprises a plurality of hollow cathode injectors disposed in fluid communication with a gas source. Each hollow cathode includes an orifice communicating with the interior space of the chamber.

According to another aspect of the present invention, an electron/plasma injector assembly is adapted for non-thermionically supplying plasma to a reaction chamber. The injector assembly comprises a main body and a plurality of replaceable or interchangeable gas nozzles. The main body has a generally annular orientation with respect to a central axis, and includes a process gas

section and a cooling section. The process gas section defines a process gas chamber and the cooling section defines a heat transfer fluid reservoir. The gas nozzles are removably disposed in the main body in a radial orientation with respect to the central axis and in heat transferring relation to the heat transfer fluid reservoir. Each gas nozzle provides fluid communication between the process gas chamber and the exterior of the main body.

According to yet another aspect of the present invention, a method is provided for depositing a sputtered material at a high deposition rate. A negatively-biased target cathode including a target material is provided in a sealed chamber. A substrate holder is provided in the chamber and spaced at a distance from the target cathode. An operating voltage is applied to the target cathode to produce an electric field within the chamber. A magnetron assembly is provided in the chamber to produce a magnetic field within the chamber. A negatively-biased, non-thermionic electron/plasma injector assembly is provided between the target cathode and the substrate holder to create an intense plasma proximate to the target cathode. A background gas is introduced into the chamber to provide an environment for generating a plasma medium. A portion of the target material is sputtered and transported through the plasma medium toward the substrate holder.

According to still another aspect of the present invention, a metal nitride material such as aluminum nitride, gallium nitride, or a related compound is produced according to the method disclosed herein. Ultra-high growth rates of approximately 0.05 μm/min to approximately 10 μm/min, diameters from approximately 1 inch to approximately 8 inches, and a thickness of at least approximately 1 mm or greater, can be achieved.

It is therefore an object of the present invention to provide a novel sputter material transport device capable of ultra-high deposition and growth rates.

It is another object of the present invention to provide a transport device capable of growing both high-purity bulk crystals and thin films having nearly bulk properties and which can be either metallic, semiconducting or dielectric materials.

It is yet another object of the present invention to provide a transport device characterized by lowered processing pressure and higher ionization efficiency.

It is still another object of the present invention to provide a transport device that operates without contamination.

Some of the objects of the invention having been stated hereinabove, other objects will become evident as the description proceeds when taken in connection with the accompanying drawings as best described hereinbelow.

Brief Description of the Drawings

Figure 1 is a schematic view of a conventional sputter transport device known in the art;

Figure 2 is a perspective view of a circular magnetron source known in the art;

Figure 3 is a schematic view of a hollow cathode-enhanced sputter transport device known in the art;

Figure 4 is a schematic view of a novel sputter transport device according to one embodiment of the present invention;

Figure 5A is a top plan view of an electron/plasma injector assembly provided according to one embodiment of the present invention;

Figure 5B is a cut-away vertical cross-sectional view of the injector assembly illustrated in Figure 5A taken along line 5B-5B thereof;

Figure 6 is a plot of electron injector ring current versus magnetron current exemplifying performance of the transport device illustrated in Figure 4;

Figure 7 is a schematic view of a novel sputter transport device according to a further embodiment of the present invention;

Figure 8 is a perspective view of an electron/plasma injector assembly according to another embodiment of the present invention;

Figure 9 is a top plan schematic view of the injector assembly illustrated in Figure 8;

Figure 9A is a vertical cross-sectional view of the injector assembly illustrated in Figure 9 taken along line 9A-9A thereof;

Figure 9B is a vertical cross-sectional view of the injector assembly illustrated in Figure 9 taken along line 9B-9B thereof;

Figure 10A is another perspective view of the injector assembly illustrated in Figure 8;

Figure 10B is a top plan view of the injector assembly illustrated in Figure 8;

Figure 11 is a perspective view of the injector assembly illustrated in Figure 8 showing the operation thereof and an exemplary electron/plasma injection pattern;

Figure 12 is a plot comparing the source performance of a transport device provided according to the present invention and that of a conventional magnetron source;

Figure 13 is a perspective view of a rectangular magnetron source which can be employed in combination with the present invention;

Figure 14 is a schematic view of a novel sputter transport device according to an additional embodiment of the present invention; and

Figure 15 is a schematic view of a novel sputter transport device according to a yet another embodiment of the present invention.

Detailed Description of the Invention

Referring now to Figure 4, a sputter transport device generally designated **100** is illustrated according to one embodiment of the present invention. Key operating components of transport device **100** are contained within a grounded, sealable sputter-transport chamber **102**. As will be appreciated by persons skilled in the art, a pumping system (not shown) is provided to control the pressure (vacuum or otherwise) within chamber **102**. Supply systems (not shown) are also provided for delivering a background gas (e.g., argon), and a reactive gas (e.g., nitrogen) in the case of reactive sputtering, into chamber **102**. In some applications of the present invention, the reactive gas may also serve as the background gas.

A cathode **104** constructed from a metallic, dielectric, or compound target material is bonded to a target holder **106** to establish thermal contact therebetween. Target cathode **104** may be provided in the form of a circular disk or a rectilinear plate, or may have some other shape. Target holder **106** is preferably constructed of copper or other relatively inexpensive material that offers acceptable levels of both thermal and electrical conductivity. A heat exchanger system (not shown) is provided to circulate a heat transfer medium such as water through target holder **106** to keep target holder **106** (and thus

target cathode **104**) cool. In other embodiments, a heater (not shown) can be provided to heat target holder **106**. A magnetron assembly **110** includes a set of oppositely-poled magnets **112** and **116** connected by a magnetic field return plate **118**. The arrangement of magnets **112** and **116** preferably constitutes a central magnetic bar **112** surrounded by an outer magnetic annulus **116**, although other arrangements and shapes could be provided. Magnets **112** and **116** are preferably located on the side of target holder **106** opposite to target cathode **104**. If desired, a conventional cooling system (not shown) can be provided to cool magnets **112** and **116**. A negative bias voltage is applied to target holder **106** by connecting target holder **106** in series with a voltage source **120**.

A substrate holder **130**, which serves as the primary anode, is disposed in chamber **102** in parallel with and spaced at a distance from target cathode **104**. Preferably the spacing is in the range of approximately 2 cm to 20 cm. Substrate holder **130** can be constructed from any material that is either electrically conductive or isolated, and can be provided as either a cooling structure or a heating structure. It is preferable that transport device **100** be oriented such that target cathode **104** is physically situated opposite to substrate holder **130**, but can be either vertically above or below substrate holder **130**. A substrate **132** is disposed on substrate holder **130**. Depending on the specific application of transport device **100**, substrate **132** can be either initially provided in bulk form on which a thin-film is to be deposited, or it represents the growing bulk material grown through use of transport device **100**.

As will be appreciated by persons skilled in the art, substrate holder **130** or an associated transfer arm (not shown) can be used to transport substrate

holder **130** and, if applicable, an initially-provided substrate material into and out from chamber **102**. In addition, a load lock or similar component (not shown) can be provided to serve as an interface between chamber **102** and the ambient environment to assist in maintaining reduced pressure in chamber **102** when substrate holder **130** and/or an initially-provided substrate material is loaded and thereafter removed from chamber **102**. Other known processing components can used as appropriate to assist in implementing the methods of the invention involving the use of transport device **100**, including an electronic control system, a power supply system, a pressure monitoring system, a mass flow control system, a temperature monitoring system, and a system for automated tracking and transport of workpieces.

As one key aspect of the present invention, an injector assembly generally designated **150** is disposed in chamber **102** proximate to target cathode **104**, and is separately, negatively biased through its serial connection with a voltage source **152**. Hence, injector assembly **150** serves as a cathode apart from and additional to target cathode **104**, such that transport device **100** can be characterized as being a triode sputtering source.

Referring to Figures 5A and 5B, injector assembly **150** includes a plurality of injectors **152** serving essentially as individual hollow cathodes. Each injector **152** terminates in an inlet orifice **152A** communicating with the interior of chamber **102** in the region proximate to the surface of target cathode **104**. In the present embodiment, injector assembly **150** takes the form of an injector ring such that each inlet orifice **152A** faces radially inwardly with respect to chamber **102**, although individual injectors **152** can be arranged in a linear or other suitable configuation.

In operation, electrons in the form of supplemental or auxiliary plasma beams are non-thermionically emitted from injectors **152** as a result of the increase in electric field strength at these points, such that the electrons are subsequently injected and coupled into the gradient of the magnetic field (represented by virtual field lines **B**) established by magnetron source **110** to generate an intense plasma. Injector assembly **150** may thus be characterized as a cool, non-thermionic electron/plasma source which injects an approximately equal number of ions and electrons into the region illustrated in Figure 4 proximate to target cathode **104**, thereby creating a higher probability of ionization of the target material. Figure 6 is a graph of magnetron current at a constant applied voltage as a function of injector ring emission current. The slope of the curve indicates a measure of electron coupling into the magnetron discharge. An increase in magnetron current is observed due to the added electrons from injector assembly **150**. This effect can be seen as a significant increase in the plasma brightness, as well as a significant increase in the sputter deposition rate. The intense plasma created in the proximity of the surface of target cathode **104** results in the significant increase in deposition rate by more than ten times over conventional techniques. Injector assembly **150** also serves to electrostatically confine the plasma to form a broad plasma beam **160** directed toward substrate **132**. Due to the bulk mass and/or cooling design of injector assembly **150**, its temperature remains low and accordingly no thermionic emission, evaporation or contamination takes place during deposition.

Transport device **100** can be operated in either continuous DC, pulsed DC, AC or RF mode, which enables transport device **100** to reactively sputter a wide range of both conductive and insulating materials at very high rates. Due to

the high percentage of gas ionization, the material of target cathode **104** is sputtered at ultra-high rates sufficient to prevent a detrimental insulating layer from forming on the target surface. In addition, due to the very high ion energies associated with the process according to the present invention, large amounts of material can be sputtered. Device **100** has been proven to operate successfully in 100% reactive gas environments, therefore demonstrating the stability of the device under very reactive conditions.

As described above, a negative bias is applied to target holder **106**, which generates a magnetron sputtering discharge, and a separate negative bias is applied to injector assembly **150**. This generates a very intense plasma, with beamlets of plasma emitting from each injector **152** of injector assembly **150**. The added plasma density and ionization percentage in the region of the target cathode **104** increase the amount of target bombardment, thereby causing increased sputter rates. Due to the increased utilization of sputtering gas, the background processing pressure can be lowered from, for example, approximately 5 mTorr to approximately 0.1 mTorr, which can improve the microstructural properties of materials being formed. This pressure decrease increases the mean free path of molecules, enabling the creation of plasma beam **160** between target cathode **104** and substrate holder **130** (i.e., the anode) which is characterized by very high ionization efficiency and achievement of ultra-high sputter transport rates.

Referring to Figure 7, a sputter transport device, generally designated **200**, is illustrated according to another embodiment of the present invention. In this particular embodiment, a biased containment shield **202**, constructed from aluminum or other conductive material, is disposed in chamber **102** between

target cathode **104** and substrate holder **130** and is surrounded by a containment magnet or magnets **204**. A high voltage applied to containment shield **202** from a voltage source **206** acts to focus the sputtered material and plasma beam **160** onto the growing substrate **132**, thereby increasing the transport efficiency of the sputtered material (such as aluminum nitride) to substrate **132**. Ions and electrons become trapped within the containment region under the influence of the electric and magnetic fields and subsequently deposit on substrate **132**.

Under some circumstances, the user of transport device **100** or **200** might find that the heating of injector assembly **150** causes low-melting-point metals to melt. This problem can be overcome by cooling injector assembly **150** with a copper cooling ring **220**, which is also illustrated in Figure 7.

Referring to Figures 8-11, a preferred embodiment of a fluid-cooled, ring-shaped injector assembly generally designated **300** is illustrated. Injector assembly **300** includes a main body **302** and an outer collar **304** removably secured by clamping screws **306**. Main body **302** includes a process gas section **302A** and a cooling section **302B**. As best shown in Figures 9A and 9B, process gas section **302A** and outer collar **304** together define a process gas chamber **308**. Individual injectors for supplying electrons and cool plasma, indicated by the reference numeral **310**, are defined by interchangeable gas nozzles **312** fluidly communicating with process gas chamber **308** at one end and with sputter-transport chamber **102** at the other end. Gas nozzles **312** may or may not be constructed from the same material as target cathode **104** and/or containment shield **202**. Cooling section **302B** of main body **302** defines a cooling reservoir **314** adapted to circulate a heat transfer fluid such as water in

close proximity to each gas nozzle **312**. The heat transfer fluid is circulated through cooling reservoir **314** by means of a heat transfer fluid inlet conduit **316** and outlet conduit **318**. Process gas such as diatomic nitrogen or argon is supplied to injector assembly **300** by means of a process gas conduit system **320** that communicates with one or more process gas inlets **322** on main body **302**. Figure 11 illustrates one example of an emission pattern of plasma/electrons **310** obtainable by injector assembly **300**. The pattern as well as the gas nozzle pressure can be altered by blocking one or more of individual gas nozzles **312**.

Traditionally, sputter-deposited films have been plagued with low reactive sputter rates, excessive stress, and poor crystalline growth. Due to the non-contaminating nature of transport device **100** or **200**, however, the hollow cathode effect can be advantageously utilized to produce both single-crystal and highly-oriented polycrystalline, bulk-form substrates at lower pressures, ultra-high deposition rates, and with minimal material stress. Transport device **100** or **200** is also capable of growing epitaxial layers on substrates. Examples of deposited materials include binary, tertiary, and quaternary Group III nitride based compounds such as aluminum nitride, gallium nitride, indium nitride, aluminum gallium nitride, indium gallium nitride and aluminum indium gallium nitride, and alloys thereof. Suitable dopants can be added during the growth process. Both single-crystal and polycrystalline morphologies are obtainable. In one specific example, transport device **100** or **200** is capable of growing aluminum nitride purer than that made by powder processing methods and faster than CVD methods. Moreover, because transport device **100** or **200** exhibits a very high degree of sputter particle ionization, transport device **100** or **200** produces a

plasma beam environment that facilitates the synthesis of nitride based materials. The material grown by transport device **100** or **200** exhibits the bulk properties of nitrides due to the resulting high crystallinity and purity. In particular, bulk aluminum nitride produced from transport device **100** or **200** has a high IR and UV transmittance, a high thermal conductivity, and a high degree of c-axis orientation.

In addition to growing the materials described hereinabove, transport device **100** or **200** can be utilized to grow a variety of ceramic thin films such as aluminum oxide and zinc oxide, or to deposit copper or other metallic interconnects onto patterned electronic devices. The high transport rate also enables the high-throughput coating of objects.

Figure 12 demonstrates the dramatic improvement in deposition rate by plotting plasma current as a function of applied source voltage with transport device **100** operating under a 0.7A electron enhancement (i.e., with the inventive injector ring installed and supplying current from hollow cathode-type structures), as compared to a typical magnetron sputtering device without any electron enhancement.

Conventional planar magnetron designs suffer from poor target-material utilization because of a trenched erosion pattern that tends to form on the surface of the target material in the vicinity of the **E x B** drift path of the beam electrons. The radial narrowness of this trench results from radial compression of the plasma, which is in turn caused by the well-known "magnetic-mirror" effect. The electrons of the plasma are forced away from both small and large magnetron radii at the sites where the magnetic field converges toward the magnetic pole pieces. The electrons are compressed by these mirrors toward

an intermediate radius where the magnetic field is uniform. Both the plasma and the ion bombardment are most intense in the region of magnetic field uniformity. The magnetic-mirror effect can be reduced somewhat by designing a flatter magnetic field or by mechanically scanning the magnets back and forth during sputtering. The non-uniformity of film thickness resulting from plasma compression can be avoided by moving the substrates around during deposition. One simpler, geometric approach to improving uniformity is illustrated in Figure 13, wherein a rectangular magnetron generally designated **410** is utilized. With the rectangular geometry, the many of magnetic field lines B are situated along linear directions, and the beam electrons follow an oblong or "racetrack" **E x B** drift path at target cathode **104**. The rectangular magnetron shape can be employed in connection with the present invention if non-uniformity becomes problematic.

Localization of the plasma over target cathode **104** by the transverse magnetic field of magnetron assembly **110** results in a much lower plasma density over the substrate **132** than in the case of the non-magnetron planar diode, and ion bombardment flux to substrate **132** is reduced accordingly. This is desirable when the neutral sputtered particles alone carry sufficient kinetic energy to optimize film structure, or when it is important that the substrate heating that results from ion bombardment be kept to a minimum. In other cases, however, it might be desirable to further increase film bombardment while retaining the low operating pressure of the transport device **100** or **200**. One method for increasing ion bombardment of the growing film is to "unbalance" the magnets of magnetron assembly **110**, such as by downsizing central magnet **112** such that the central magnet **112** cannot pull in all the field lines emanating

from outer magnets **116**. Hence, in the unbalanced configuration, the magnetic field lines that are not pulled into central magnet **112** will curve away toward substrate holder **130**. Because electrons traveling parallel to a magnetic field are not influenced by the magnetic field, they can escape along these wayward field lines and travel toward substrate **132**. The escaping electrons pull positive ions along with them by ambipolar diffusion and hence increase ion-bombardment flux to substrate **132**. In addition, the bombardment energy can be increased by negatively biasing substrate **132**.

Another way to increase ion-bombardment flux to the growing film is to provide an RF-powered coil to ionize the mostly neutral sputtered-particle flux during transport to substrate **132**. The coil operates by coupling energy inductively into a secondary plasma downstream of the magnetron plasma.

Referring now to Figure 14, a sputter transport device, generally designated **600**, is illustrated according to an additional embodiment of the present invention. Many of the components of sputter transport device are similar to those of sputter transport device **100** shown in Figure 4. In particular injector assembly **150** as described above is utilized to enhance the material transport process. A primary difference is that a liquid target **604** such as liquid-phase aluminum or gallium is provided as a source species. The target holder in this embodiment is provided in the form of a cup **606** to contain the liquid target material. Preferably, this target holder should be constructed from a material suitable for withstanding the heat involved and which will not contaminate the target material. Candidate materials for target holder **606** include molybdenum and stainless steel. In one embodiment, a 6" diameter molybdenum liquid gallium or aluminum target holder **606** is employed to prevent reaction of the

holder with a high purity (99.9999%) liquid gallium or aluminum source **604**. In order to obtain a flat uniform liquid surface of the gallium or aluminum, sufficient wetting of the gallium or aluminum to the molybdenum holder **606** must occur. To this end, grooves can be cut into the bottom of target holder **606** to increase its surface area and thereby increase its wettability. In addition, a breathing hole connecting the grooves can be provided to eliminate any gas trapped under the liquid gallium or aluminum.

Referring now to Figure 15, a sputter transport device, generally designated **700**, is illustrated according to another embodiment of the present invention. Sputter transport device **700** is equipped with a biased containment shield **202** and containment magnets **204**, similar to those described in reference to Figure 7. A high voltage applied to containment shield **202** will focus the sputtered material onto growing substrate or film **132**, thereby increasing the transport efficiency of Ga or Al to substrate or film **132**.

Sputter transport devices **600** and **700** operate as described above. Gallium (or aluminum) particles sputtered from the cathode react with atomic nitrogen in the cathode magnetic fields. The gallium nitride (or aluminum nitride) particles travel through the containment magnetic field to the substrate. The quality of growth material is determined by the nucleation and growth at the substrate surface.

It will be understood that various details of the invention may be changed without departing from the scope of the invention. Furthermore, the foregoing description is for the purpose of illustration only, and not for the purpose of limitation—the invention being defined by the claims.